IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	Robert L. Goldsmith	Paper No.:	
Serial No.:	10/676,671	Examiner:	Menon, Krishnan S.
Filed:	10/1/2003	Group No.:	1723
For:	Membrane Devices with Controlled Transmembrane Pressure and Method of Use	Docket No.:	19642-00019

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

CERTIFICATE OF ELECTRONIC FILING

I hereby certify that this Declaration is being electronically filed this 4th day of April, 2007, and goes to Mail Stop Amendment, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

Brian M. Dingman

DECLARATION OF ROBERT L. GOLDSMITH UNDER 37 CFR 1.132

Robert L. Goldsmith declares as follows:

1. I am a named inventor in the subject application. I have a PhD in chemical engineering granted from the Massachusetts Institute of Technology in 1966. I have worked in the field of ceramic membrane devices for the last 20 years. Due to my education, long experience, and patents in the field (including patents 4,983,423; 5,009,781; 5,106,502; 5,108,601; 5,114,581; 5,120,576; 5,221,484; 5,879,715; 6,126,833; 6,695,967; and 6,767,455), I believe that I am an expert in the field of ceramic membranes and their uses.

2. I have reviewed the January 17, 2007 Office Action, about which I have the following comments.

3. Commercialization of the Membrane Monolith Structure covered by USP 4,781,831

It is relevant to present an overview of the commercial history of monolith membrane devices made under this patent, as it shows the knowledge of this technology has been widespread in the field.

a. CeraMem Separations, LLP began selling devices of the disclosed structure ca. 1993. About 20 commercial installations were made, throughout the world (US, several European countries, Japan, India, etc.). The equity in CeraMem Separations was sold to Corning, Inc. in 1997, and CeraMem Separations ceased selling the products then. Product literature for the CeraMem Separations, Inc. products (published prior to mid-1997 when all sales ceased) and products photographs are attached as Exhibit A. These documents were widely distributed throughout the world.

b. Corning, Inc. Sales of products covered by this patent were sold by Corning, Inc. after 1997 under the Trademark "Cercor". Product literature of Cercor (dated 1999) was widely distributed through out the world and is attached as Exhibit B. Sales were made for over forty installations.

c. CeraMem Corporation began selling a new version of products made under this patent in 2002. A undated product bulletin is attached as Exhibit C. First commercial sales occurred in 2003, and about 25 commercial installations have been made. A membrane photograph is appended as Exhibit D.

d. NGK Insulators, Ltd began selling a product under this patent ca. 2000, and definitely by 2002. CeraMem Corporation has "royalty" reports showing sales in 2002. An announcement of NGK's product (found at http://www.ngk.co.jp/english/news/2001/0213.html) is attached as Exhibit E. A photograph of the NGK product (found at http://www.ngk.co.jp/english/news/2003/1014.html) is attached as Exhibit F.

Hence, membrane modules of the structure disclosed in USP 4,781,831 have been sold commercially for at least 14 years by four separate corporate entities.

4. Examples of Patents For Membrane Modules for Controlled Transmembrane Pressure

The following patents show the early recognition of the benefits of controlled transmembrane pressure by use of permeate circulation co-current with feed flow in membrane module modules of constructions highly different from those in the present invention.

US Patent 4,105,547: Filtering process (filed 1975)

US Patent 4,906,362: Arrangement in membrane filter (filed 1989)

US Patent 5,906,739: Membrane filtration assembly (filed 1996)

These patents have claims covering permeate circulation in specific membrane module constructions. None of the patents suggests in any way applying this technique to a multichannel monolith membrane device.

Other patents cited in the present application 10/676,671 disclose attaining controlled transmembrane pressure by a gradient in membrane element resistance from the feed end to the retentate end of the element.

These patents show the recognized need for membrane module designs for liquid phase separations with controlled transmembrane pressure.

5. Overview of Bactocatch Process.

The excerpt of a recent paper by Barauh, et al. (found at http://www.aseanbiotechnology.info/Abstract/21018510.pdf) and attached as Exhibit G, describes the controlled transmembrane Bactocatch process. The references indicate the early commercialization of this process. Note, in particular, Figure 1 and ref. 5 from 1979 (US Patent 4,140,806) and ref. 6 from 1974 (SW 74,16,257). This Bactocatch process uses co-current permeate circulation along the length of a membrane module while feedstock flows within membrane elements along their full length in a membrane module. The devices of the prior art Rajnik patent 6,077,436, with the structure of permeate drainage ports along the length of the membrane module, are incapable of achieving this process without some further, undisclosed invention.

The control of transmembrane pressure in membrane processes has been of substantial commercial interest for about twenty years, notably in the cold pasteurization or sterilization of skim milk and beer by microfiltration. Several polymeric and ceramic membrane companies have introduced products to achieve this process, and ceramic membranes have been of special interest because of their ability to be steam sterilized and cleaned aggressively. However, the high cost of traditional prior art ceramic membranes has been a serious impediment to their market acceptance. The introduction of permeate circulation within large diameter ceramic monolith membrane devices (the subject of my invention) circumvents this cost limitation. Based on my experience, CeraMem sells its membrane modules (or projects costs once in full production) for about ¼ to ½ the price of traditional prior art ceramic membranes modules used for the applications of interest. As such, the present invention offers the benefits of ceramic membranes in a cost-effective manner heretofore unavailable.

6. Conclusions relevant to the obviousness of combining a monolith membrane device containing permeate conduits with controlled transmembrane pressure by permeate circulation.:

1. Modules of the structure of US patent 4,781,831 have been on the market commercially for 14 yrs. Knowledge of the existence of such membrane modules is widespread and known throughout the membrane industry.
2. Devices for controlled transmembrane pressure using permeate circulation have been in the patent art for at least 20 yrs. Hardware for the Bactocatch process has been sold for over 20 yrs, first by Alfa-Laval and subsequently by the acquirer of Alfa-Laval's membrane technology, Tetra Pak. GEA Filtration now sells controlled TMP systems based on use of TAMI controlled gradient membranes, in place of permeate recycle, to achieve a controlled transmembrane process.
3. The fact that monolith membranes were disclosed by me in 4,781,831 (1988), and the benefits of transmembrane pressure control need and means have been known for over twenty years without the recognition of the present application, indicates that the combination is not obvious to one "skilled in the art". No one has done this or suggested this possibility, to my knowledge, until the present patent application was filed.
4. This conclusion is especially relevant as I, as the inventor of the primary application (4,781,831), am clearly "skilled in the art" and did not make this connection until some 17 years after making the application for USP 4,781,831. During this period, I was active and fully employed in the membrane field, managing the development of ceramic membranes and membrane processes at CeraMem Corporation (the assignee of the present application) and as chairman of the board of CeraMem Separations, Inc. During this period I remained current in the membrane field art – patents, published technical literature, attendance at industry conferences, presenter of papers at technical conferences, and an author of numerous technical papers and reports on Government sponsored research programs in the field.
5. Finally, I am unaware of any patent art or commercial use of the method of using co-current permeate flow in multichannel monolith modules with permeate conduits to achieve a controlled transmembrane pressure.

The undersigned declares that willful false statements and the like are punishable by fine or imprisonment, or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon. All statements made of the declarant's own knowledge are true and that all statements made on information and belief are believed to be true.

Robert L. Goldsmith

April 14, 2007

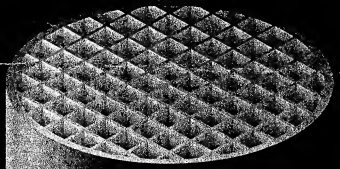
Date

Exhibit A

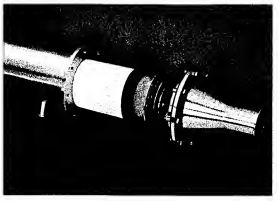
CeraMem

SEPARATIONS

Ceramic Membrane Filters



LIQUID FILTERS

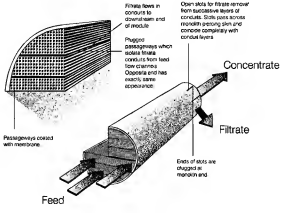


Housing/filter assembly.

LIQUID FILTRATION PRODUCTS

CeraMem manufactures and sells crossflow membrane filters with pore sizes in the Ultrafiltration (UF) and Microfiltration (MF) ranges.

CeraMem purchases ceramic monoliths from Corning Incorporated. These monoliths contain a large number of 2 mm or 4 mm square parallel passageways extending from one face to the other. CeraMem modifies the monolith support by converting some of the passageways to filtrate conduits. Ceramic membrane coatings are then applied to the remaining monolith passageways. One or more membrane layers are applied and sintered to form a strongly bonded ceramic membrane with a stable, controlled pore size. These membranes have FDA clearance for contact with food.



Monolith membrane module with filtrate conduits.

CeraMem sells complete assemblies consisting of the ceramic filter modules mounted in housings. These housings are offered in either a 3-A approved sanitary stainless steel design or a less expensive, non-sanitary industrial design. In both cases, the ceramic filter is fitted with elastomeric "boots" that fit over each of the two faces of the filter. These boots seal the permeate space to prevent contamination by the feed/concentrate.

CeraMem ceramic membrane filters for liquid separation are:

Rugged

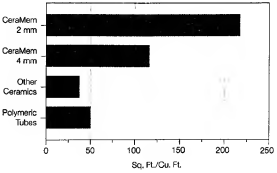
They can withstand high operating temperatures and are unaffected by oxidants, organic or hydrocarbon solvents, harsh cleaning chemicals and steam. CeraMem filters are useable in the 3-12 pH range. They can be backpulsed and backflushed to extend time between cleanings.

These features make them ideally suited for demanding applications such as clarifying organic process streams, reclaiming solvents, treating wastewaters containing solvents, recovering energy by recycling hot streams, and treating streams having high viscosities at ambient temperatures.

Compact

With their very high packing density (membrane area/module volume), CeraMem filters allow you to use fewer membrane modules, resulting in lower capital and operating costs for less piping, valving and pumping capacity.

Compactness of CeraMem Liquid Filters vs. Alternatives

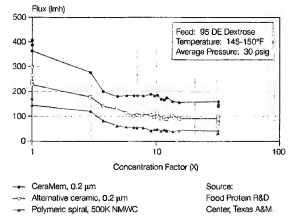


High Performance

CeraMem modules have low hold-up volumes for both feed and filtrate, minimizing losses of valuable or time-sensitive fluids within the module or housing assembly.

In many applications, the proprietary chemistries of CeraMem's membranes result in higher productivity (flux) than competitive membranes. The example shown here is for dextrose clarification.

Clarification of Dextrose Derived from Hydrolysis of Corn Starch Performance of Competing Membranes



Cost Effective

The combination of low initial cost per square foot of membrane area, lower systems and operating costs due to compactness, higher performance and long membrane lifetimes means CeraMem membrane systems are less expensive to install and operate than many polymeric systems.

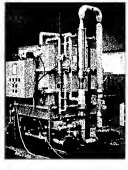
LIQUID FILTRATION APPLICATIONS

CeraMem ceramic membrane crossflow filtration products are used by the food and beverage, petroleum, chemical and petrochemical industries, as well as wastewater treatment facilities to clarify, separate and decontaminate liquids. Below is a partial list of application examples.

Replacement of Conventional Dead-ended Filtration

CeraMem filters eliminate the costs of purchase, handling, disposal and product losses associated with diatomaceous earth filtration. Microfiltration with CeraMem filters often allows recycle of streams or higher value alternative dispositions than traditional filter aids.

MF system operating upstream of RO on dairy condensate.



System operating on industrial laundry waste.

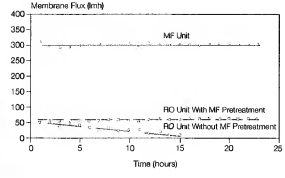
Pilot system for clarification of ethanol stillage.

CeraMem filters have successfully replaced dead-ended filtration in juice clarification, dextrose and sucrose clarification and catalyst haze removal from edible oils, among other applications.

Filtration Upstream of Reverse Osmosis

CeraMem ultrafiltration membranes, installed upstream of reverse osmosis units, can remove components that would quickly foul reverse osmosis membranes.

Evaporator Condensate Treatment by Reverse Osmosis (RO) With and Without Pretreatment by Microfiltration (MF)



Cleaning and Recycling Industrial Laundry Waste

CeraMem ultrafiltration membranes allow industrial laundries to clean hot effluents for water recycling. Because of their high temperature tolerance, these ceramic filters can be used in very hot liquid streams, saving chemical and energy costs during the recycling process.

System cleaning vehicle wash water.

Oil Removal from Wastewater Streams

CeraMem's ultrafiltration membranes will concentrate oily water emulsions, permeating the clean water for discharging or recycling and recovering an oily concentrate for disposal or recycling.

CeraMem

SEPARATIONS

Let us help you meet your liquid and gas filtration needs with our advanced line of ceramic membrane filters.

For additional information, please contact us at:

CeraMem Separations Incorporated
20 Clematis Ave.
Waltham, MA
02154
Telephone
(617) 899-0467
Fax
(617) 899-1227

Ceramic Membrane Filters

CeraMem Separations Incorporated manufactures and markets patented ceramic membrane products for environmental and process applications. Its membrane filters were first marketed in 1991 and are used worldwide to clarify, separate, and decontaminate liquids, and to remove particulate matter from exhaust-gas emissions.

The company is headquartered in Waltham, Massachusetts and maintains a sales office in London, and sales agents in Düsseldorf, Liège, Madrid, Tokyo and Seoul.

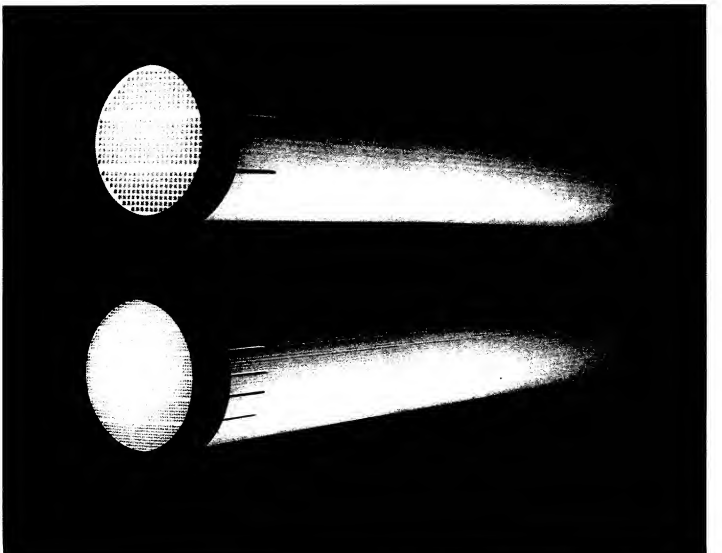
CeraMem Separations, Inc. is a joint venture of CeraMem Corporation, Exxon Corporation (through its wholly-owned subsidiary, Enjay Inc.) and Corning Incorporated.

CeraMem Corporation was founded in 1986 to undertake technical development of proprietary ceramic membranes and is a continuing source of potential new ceramic membrane technologies for CeraMem Separations.

Corning Incorporated provides CeraMem Separations with a secure supply of the honeycomb ceramic monoliths that CeraMem coats to make its products.

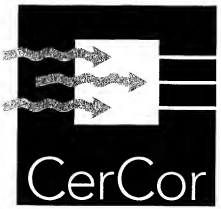
Exxon provides access to the potentially large petroleum and petrochemical markets for CeraMem's rugged, heat- and hydrocarbon-resistant membranes.

**CeraMem Filters —
Advanced Technology
for Advanced Solutions**



Photograph of CeraMem Separations, Inc. Membranes 144 mm diameter x 864 mm length

Exhibit B



SEPARATIONS

Ceramic
Membrane
Crossflow
Filters for
Liquid
Filtration

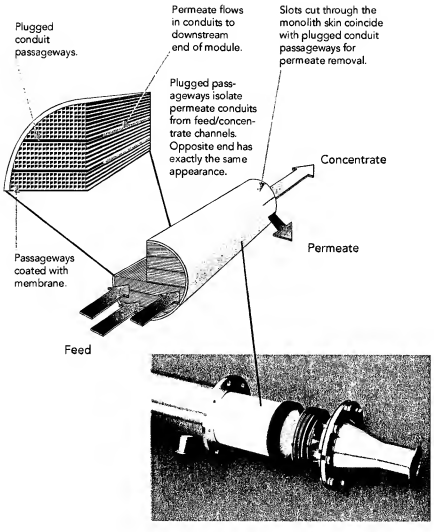
CerCor Filters

CORNING

Liquid Crossflow Filters

CerCor Separations manufacturers and sells crossflow membrane filters with pore sizes in the Microfiltration (MF) and Ultrafiltration (UF) ranges.

These filters contain a large number of approximately 2mm square parallel passageways extending from one face to the other. A CerCor patented approach modifies the monolith support by converting some of the passageways to permeate conduits. This enables the entire filter diameter to be effectively utilized.



Ceramic membrane coatings are then applied to the remaining monolith passageways. One or more membrane layers are applied and sintered to form a strongly bonded ceramic membrane with a stable, controlled pore size. These membranes have FDA clearance for contact with food.

CerCor sells complete filter assemblies consisting of the ceramic filter elements mounted in housings. These housings are offered as either a 3-A approved sanitary stainless steel design, or a less expensive, non-sanitary industrial design. In both cases, the ceramic filter is fitted with elastomeric "boots" that fit over each of the two faces of the filter. These boots seal the permeate space to prevent contamination by the feed/concentrate.

CerCor ceramic membrane crossflow filters for liquid separation are:

Rugged

They can withstand high operating temperatures and are unaffected by oxidants, organic or hydrocarbon solvents and steam. CerCor filters are usable in the 2-13 pH range. They can be backpulsed and backflushed to extend time between cleanings.

These features make them ideally suited for demanding applications such as clarifying organic process streams, reclaiming solvents, treating wastewaters containing solvents and recovering energy by recycling hot streams.

CerCor
SEPARATIONS

Compact

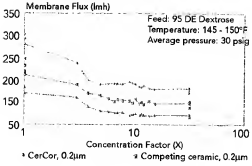
With their very high packing density (membrane area/module volume), CerCor filters allow you to use fewer membrane modules. This results in lower capital and operating costs as a result of decreased piping, valving and pumping requirements.

High Performance

CerCor filter assemblies have low hold-up volumes for both feed and permeate, minimizing losses of valuable or time-sensitive fluids within the filter assembly.

In many applications, the proprietary chemistries of CerCor's membranes result in higher productivity (flux) than competitive membranes. The example shown here is for dextrose clarification

Clarification of Dextrose Derived from Hydrolysis of Corn Starch Performance of Competing Membranes



CerCor ceramic membrane cross-flow filtration products are used by the food and beverage industries, as well as wastewater treatment facilities to clarify, separate and decontaminate liquids.

Cost Effective

The combination of low initial cost per square foot of membrane area, lower system and operating cost due to compactness and higher performance, make CerCor membrane systems less expensive to install and operate than alternative ceramic and many polymeric systems.

Liquid Filter Applications

Juice Clarification & Filtration

CerCor microfiltration membranes are used in juice clarification applications removing suspended solids without stripping color. Long term stable flux rates allow several days between required cleanings in many applications.

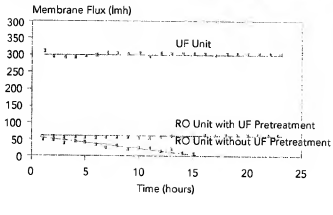
Oil Removal from Wastewater Streams

CerCor's ultrafiltration membranes will concentrate oily water emulsions. Clean water permeate is discharged, oily concentrate is collected for disposal or recycling.

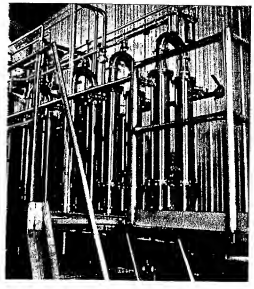
Filtration Upstream of Reverse Osmosis

CerCor ultrafiltration membranes, installed upstream of reverse osmosis units, can remove components that would quickly foul reverse osmosis membranes.

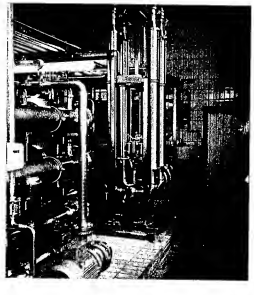
Evaporator Condensate Treatment by Reverse Osmosis (RO) With and Without Pretreatment by Ultrafiltration (UF)



MF system for juice clarification and filtration



UF system operating upstream of RO on dairy condensate



CerCor Filters Advanced Technology for Advanced Solutions

CerCor Separations manufactures and markets patented ceramic membrane filter products for environmental and process applications. Its membrane filters were first marketed in 1991 and are used worldwide to remove particulate matter from gas and liquid streams.

The company is headquartered in Corning, NY.

For additional information, please contact us at:

CerCor Separations Business
Corning Incorporated
SP-YE-01-02
Corning, New York 14830
Tel: (607) 974-2514
Fax: (607) 974-5713
E-Mail: Cercor@corning.com

Corning International
Corning GmbH
Abraham-Lincoln-Str. 30
D-65189 Wiesbaden/Germany
Tel: +49 611 7366-114/-100
Fax: +49 611 7366-143
E-Mail: CIGermany@corning.com

CerCor
SEPARATIONS

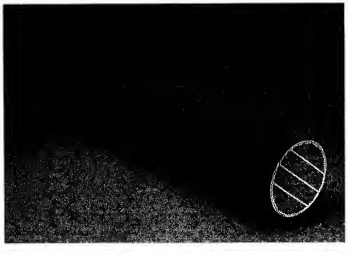
Exhibit C

New Generation Ceramic Membranes

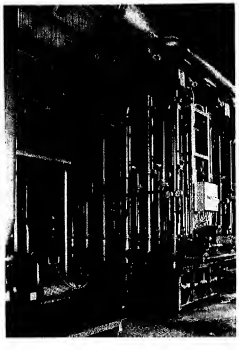
Production Scale Modules

This new generation of 'honeycomb' inorganic membrane modules for microfiltration and ultrafiltration has a variety of *abrasion-resistant* ceramic membranes applied to silicon carbide monolith supports. Product features include

- Compactness
- Low cost
- High temperature capability
- Chemical inertness



Full Size Membrane Element with 115 ft^2 (10.7 m^2) Membrane Area



System with 42 Modules

Many industrial plants totaling tens of thousands of square feet of membrane have been sold in countries around the World using this patented technology in "first generation" CeraMem™ products. This "second generation" CeraMem membrane module incorporates new chemically inert membranes and support monoliths (silicon carbide) and features superior chemical and mechanical durability. The membrane elements are extremely compact with > 230 ft^2 membrane area/ft^3 (> 760 m^2 membrane area/m^3).

CeraMem

Production Scale Membrane Module Specifications

Membrane Elements

Membrane elements are 34 inches (864 mm) long and 5.6 inches (142 mm) in diameter. Membrane area is approximately 115 ft^2 (10.7 m^2). Single monolith membrane elements with membrane area up to 410 ft^2 (38 m^2) are planned.

Membrane pore sizes and chemistries available:

Product Code	Membrane Type	Water Flux (Typ.), lmh/bar
PM-0200-A	0.2 μm - α-alumina MF	400
PM-0100-A	0.1 μm - α-alumina MF	350
PM-0010-T	300,000 MWCO - titania UF	250
PM-0005-S	50,000 MWCO - silica UF	200

Ongoing research at CeraMem is expected to result in the expansion of the product line to include nanofiltration, pervaporation, and gas separation membranes, all with inorganic membranes.

Operating Conditions:

Operating Parameter	Range
Maximum Temperature	130°C, dependent upon seals
Maximum Trans-Membrane Pressure	150 psi (10 bar)
pH Range (PM-0200/0010-A & PM-0010-T)	2 - 13
(PM-0005-S)	2 - 9 [1]
Recommended Cross Flow Velocity	9 - 12 ft/sec (3-4 m/sec)
Volumetric Flow-Rate for 12 ft/sec	300 gpm (70 m^3/hr) [2]
Pressure Drop at 12 ft/sec	15 psi (1.2 Bar), H_2O @25°C.

[1] Highly hydrophilic, non-fouling membrane for non-alkaline service
[2] At 12 ft/sec crossflow velocity, corresponds to 0.023 theo. hp/ft^2 (at 3.7 m/s, 0.18 theo. kW/m^2)

Applications Recommended:

Tests to date indicate that there is little restriction on the use of these membranes for any conventional application. These membranes may be used for many applications in the food, dairy, beverage, pharmaceutical, metal working and finishing and many other industries.

For additional information, contact:

CeraMem Corporation
12 Clematis Avenue, Waltham, MA 02453 www.ceramem.com
Tel. (781) 899-4495 Fax (781) 899-6478 Email sales@ceramem.com

Exhibit D

Photograph of CeraMem Corporation Membrane 144 mm diameter x 864 mm length

Exhibit E

NGK Expands Water Treatment Plant Business Through Large Ceramic-Membrane Water-Purification Systems

NGK INSULATORS, LTD. (President: Masaharu Shibata, Head Office: Nagoya, Japan) has developed a next-generation water-purification technology using large ceramic-membranes, and is enlarging its water treatment engineering business.

In the 1960's, NGK entered the water purification industry by offering ceramic filter underdrains for rapid filtration ponds. Since then, the company has been expanding its business as a specialized materials/equipment supplier. From 1989, the company started development of water purification systems using ceramic membranes, and from 1991 to 1993, participated in the "MAC 21 Project" - a project sponsored by Japan's Ministry of Health and Welfare for research and development of water purification systems using membranes. In 1996, NGK developed a commercial ceramic-membrane water-purification system for the first time in Japan, and has been selling these systems to small-scale purification facilities for public water systems.

Currently more than 96% of Japan is served by a public water supply, therefore development and introduction of new technology is increasing for the remodeling and renewal of these facilities. NGK newly developed the large ceramic-membrane water-purification system while targeting large savings in cost and space. The company plans marketing efforts to stimulate orders from middle- and large-scale purification plants, and is also supplying ceramic membrane units to water-related machinery and equipment manufacturers. Responding to the changing market, NGK will grow its businesses to establish a position as a major water treatment engineering company. In FY2005, the company aims at 10-billion yen in sales of water treatment facilities, primarily those utilizing the ceramic-membrane purification system.

NGK Ceramic Membrane Water Purification Systems

Monolithic and internal-pressure type ceramic-membrane elements of 180mm diameter, 1000mm length and 15m^2 membrane surface area are employed in the newly developed system. A goal of NGK's development of this new system is large savings of cost and space compared to conventional water purification facilities using ceramic-membrane elements of 30mm diameter and 1000mm length. Micro filtration through 0.1 micron size pores and highly porous ceramic membranes enable complete elimination of turbidity and impurities such as colloids and bacteria contained in raw water.

Characteristics of Ceramic Membrane Elements

1. No chemical degradation and also no deterioration from heat and pressure enable longer operation.
2. Superior thermal/acid resistance and no dissolution of impurities.
3. High mechanical strength prevents damage to the membranes.

4. Superior chemical corrosion resistance allows easy recovery of membrane performance through chemical cleaning compared to polymeric membranes.
5. Used membranes can be recycled as ceramic material for other uses.

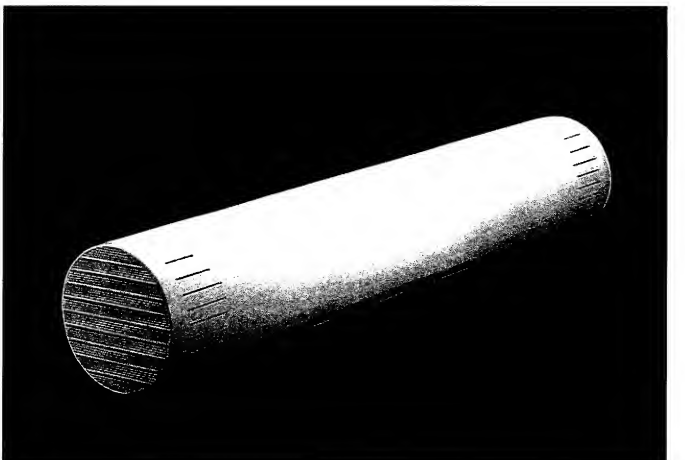
Characteristics of Ceramic Membrane Water Purification System

1. Enables stable treatment capacity for fluctuating raw water turbidity.
2. Pre- and mid-chlorination is unnecessary, so that generation of trihalomethanes is reduced.
3. Since sedimentation and rapid filtration ponds are not necessary, smaller construction space is possible.
4. Fully automated system, including backwash process, enables unattended operation.
5. Long-life ceramic membranes allow less-frequent replacement of membranes.
6. Dead end filtration enables high recovery rate of water (more than 98%).
7. Small running cost per filtration unit (below 10 yen/m^3).
8. Automated treatment process allows simplified maintenance.

In July 2000, the large ceramic-membrane module received a certification by the Association of Membrane Separation Technology, and the large ceramic-membrane water-purification system was certified by the Japan Water Research Center in November 2000.

Ceramic membrane elements

Exhibit F



NGK Insulators Ltd Membrane (2003), 180 mm diameter x 1000 mm length

http://www.ngk.co.jp/english/news/2003/1014.html

Exhibit G

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Scale-up from laboratory microfiltration to a ceramic pilot plant: Design and performance

Gautam Lal Baruah [1], Arpan Nayak, Georges Belfort *

Howard P. Isermann Department of Chemical and Biological Engineering, Rensselaer Polytechnic Institute, Troy, NY 12180, USA

Abstract

A highly instrumented pilot microfiltration (MF) plant featuring a ceramic microfiltration membrane, back-pulsing device, permeate re-circulation in co-flow to achieve uniform transmembrane pressure (UTMP) and a cooling/temperature control system was designed, built and tested successfully with transgenic whole goat milk (TGM). In a previous study, it was shown that >90% of heterologous IgG could be recovered from TGM in the permeate by adopting close process control with low UTMP and operation at the iso-electric point (*pI*) of the target IgG. Here, these concepts were successfully employed to recover >90% of another IgG (*pI* = 7.1–7.5) from TGM by MF at the pilot-scale. The pilot plant MF system gave 90% yield for the same target protein with a 50% higher permeation flux rate (47 lmh instead of 32 lmh) and increased protein transmission (70% instead of 46%) as compared with the laboratory-scale MF unit. Thus, this study demonstrated the efficacy of the pilot MF plant and reinforced the generality at larger scale of the optimization methodology reported earlier by us.

Keywords: Microfiltration; Transgenic milk; Ceramic membrane; Scale-up; Design

1. Introduction

Since the 1980s, MF has been investigated as a competing technology to centrifugation for clarification and bacteria removal of milk and whey [1–3]. However, available polymeric membranes such as polysulfone, poly(ether sulfone) and polycarbonate, were not ideal in terms of chemical stability to NaOH, Na-hypochlorite and HNO_3 which form a part of the typical cleaning regimen used in the dairy industry [4]. The advent of ceramic membranes provided an excellent opportunity in terms of chemical and thermal stability as they can withstand a pH range from 0.5 to 13.5 and temperatures over 100 °C. From the standpoint of filtration, it is important to note that these membranes are quite hydrophilic, resulting in lower protein adhesion than hydrophobic membranes like polyolefins (polypropylene and polyethylene). Also, their pore size distribution is typically narrower than that of polymeric membranes. Ceramic membranes are, however, about an order of magnitude more expensive than polymeric membranes. Early processes involving MF of dairy products with ceramic membranes employed the traditional concept of conducting MF at high inlet pressures and varying transmembrane pressure (TMP) along the membrane module [5]. This resulted in high permeation flux close to the inlet leading to cake build up and operation in the pressure-independent regime. This subsequently led to a progressive decline of protein transmission and selectivity with the progress of filtration. Hence, industrial scale-up was not feasible at that time. The problems of deteriorating MF performance and fouling were largely solved by employing the concept of low uniform TMP (UTMP) pioneered and first patented by Sandblom in 1974 [6]. UTMP operation made it possible to reap the benefit of efficient particle back-transport from the membrane wall at high axial wall shear rates (at high crossflow velocities) while maintaining low TMP in the pressure-dependent regime. As elucidated by Baruah and Belfort, in this regime, the membrane deposits are sparse and solute transport through the membrane is high [7,8]. Recent advancements have focused on alternative ways of achieving UTMP, as permeate recirculation can lead to high pumping costs and temperature rise in the system. US Filter (Warrendale, PA) has developed a module called Membralox GP, which incorporates a variation in the porosity of the

* Corresponding author. Tel.: +1 518 276 6948; fax: +1 518 276 4030.
E-mail address: belfog@rpi.edu (G. Belfort).
[1] Present address: Abbott Laboratories, AP-8, 100 Abbott Park Road, Abbott Park, IL 60064, USA.

membrane support matrix along the length of the module. A progressive increase in the porosity of the support along the length of the module has the effect of uniform permeation rates along the module. A similar concept has been employed by Tami (Nyons, France) for their Isoflux modules where the thickness of the membrane selective layer is decreased along the module length to give a uniform permeation rate. Holm et al. (Alfa Laval, Tumba, Sweden) have patented a process where UTMP is achieved by packing the module shell side with polymeric beads [9]. Another issue being tackled is the membrane packing density, which is low for tubular monotube configurations. Multichannel arrangements akin to hollow fiber bundles, adopted by Tami (France) and US Filter have alleviated, to some extent, the problem of membrane packing density [1]. Tami manufactures ceramic membrane modules with non-circular, square and lobe-shaped channels, whereas US Filter products like Membralox and Sterilox have circular channels.

Many of the advances in membrane technology described above have been fuelled by the demands and advances in MF technology as applied to the dairy industry. MF technology has a dual appeal for the dairy industry: removal of bacteria with minimal heat treatment and fractionation of proteins. The work conducted by Holm et al. [9] was supplemented by Piot et al. (see Saboya and Maubois [1]) and led to the development of the famous Bactocatch process patented by Tetra Pak [9]. As the name suggests, the idea here is to pass skim milk through a microfiltration membrane (1.4 μm) and trap bacteria in the retentate. The important parameters here are UTMP of 7.5 psi (0.5 bar) and a very high axial velocity of 7 m/s. The Bactocatch process has been adapted with a lower membrane pore size (0.1–0.2 μm) to filter whole or skim milk to produce bacteria free whey in the permeate [1]. Recently, Baruah and Belfort have combined the recommendations of the aggregate transport model (ATM) for the MF of complex polydisperse suspensions with charge-based principles and uniform axial transmembrane pressure in the pressure-dependent regime to predict and subsequently obtain excellent yields (>95% in 4 diavolumes) of chimeric IgG from transgenic goat milk [10].

In this body of research, a laboratory-scale microfiltration unit was scaled up to a ceramic pilot plant that incorporates the developments discussed above and the technology of back-pulsing [11–13]. Experiments conducted in this pilot plant have demonstrated 50% higher product sieving rates and permeation fluxes than experiments with a laboratory-scale polymer membrane unit without sacrificing high product yields in excess of 90%. The purpose of this paper is to provide details of the pilot plant design and its preliminary performance.

2. Pilot plant design

The design philosophy of the ceramic MF pilot plant envisaged a concept, which incorporates the benefits of UTMP operation, ceramic membrane material, turbulent regime mass transfer and back-pulsing. Sandblom [6] suggested the use of permeate recirculation in the shell side of the membrane module (Fig. 1a). Depending on the actual dimensions of the module, the permeate recirculation rate can be adjusted so that the pressure gradient

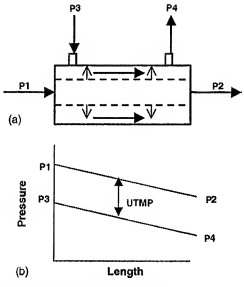

Fig. 1. Schematic of (a) UTMP membrane module with co-flow using permeate recirculation in the shell side and (b) pressure profiles for co-flow in the tube and shell sides along the length of the UTMP membrane module.

on the tube side (retentate) and the shell side (permeate) can be made approximately equal. As shown in Fig. 1b, TMP can thus be maintained at an arbitrarily low uniform value throughout the length of the module independently of the axial velocity and hence, wall shear rate in the tube. Previously, this was only possible with modules containing moving parts such as rotary disc and cylindrical modules.

This plant was designed from first principles and close coordination with vendors, GTC Biotherapeutics (Dan Couto) and industrial experts on membrane processing and technology (Robert van Reis from Genentech South San Francisco, CA; Gunnar Jonsson, Danish Technical University, Lyngby, Denmark; and Richard Mcdonnough, Memtec, San Diego, CA). The approach was to design the plant according to the methods typically employed in industry. Thus, detailed specifications (Appendix A) were written for the overall plant and its components and a piping and instrumentation diagram (Fig. 2) was made. Sizing calculations (Appendix B) for the pumps and piping were conducted based on detailed pressure drop calculations and vendor inputs on unit pressure drops. The cooling system was designed based on estimated pump recirculation heat loads. The back-pulse unit was selected based on information from US Filter, the supplier, and advice from Gunnar Jonsson. A bypass line was constructed to adjust the back-pulse amount during operation. The design of a ceramic microfiltration unit in Cornell University, Ithaca was used as a reference for vendor names for various components [14]. Details of the ceramic pilot plant with manufacturers and model numbers are given in the next section.

3. Experimental

3.1. Feed suspension

Transgenic goat milk (TGM) was supplied by GTC Biotherapeutics (Framingham, MA) from their goat farm in Charlton,

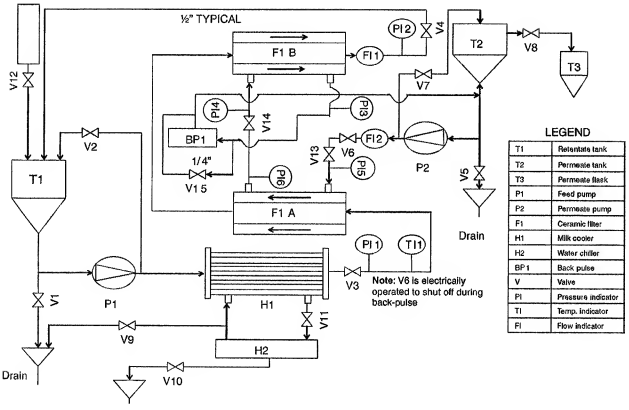

Fig. 2. Ceramic microfiltration pilot plant piping and instrumentation diagram.

MA. The human IgG concentration in the transgenic goat milk (~8 g/l) was diluted by GTC with non-transgenic goat milk to between 0.5 and 3.25 g/l so that higher volumes of TGM were made available. The IgG existed in three iso-forms and had a *pI* in the range of 7–7.5 (GTC Biotherapeutics).

3.2. *Ceramic pilot plant*

Figs. 2 and 3 show the piping and instrumentation diagram of the ceramic microfiltration pilot plant. This system consisted of three circulation loops—retentate, permeate, and cooling. The retentate loop consisted of a polished stainless steel (SS 316) retentate tank of 11 capacity (Shickel corp., VA), a rotary positive displacement variable speed sanitary pump (Model U1 018, Waukesha, IL), a miniature shell and tube cooler (Model 00455-1, Exergy, MA) and two 0.2 μm average pore size ceramic mono-tube filter modules in series (Model T1-70-75A, USF Filtrations and Separations, Fl). There were two 1.27×10^{-2} m (0.5 in.) Triclamp diaphragm valves (Model 36-2-P05-6L-E-M, Flowtech, GA) located downstream of the cooler (V3) and the second filter module (V4). V3 was included for isolating the cooler and V4 was used to vary the back pressure downstream of the filter modules. Additionally, an identical 1.27×10^{-2} m (0.5 in.) valve V2 was provided in a by-pass loop for the filters. Glycerine filled pressure indicators PI 1 and PI 2 (Model EA071010041441, Anderson Instrument Company, NY) downstream of the cooler and the second ceramic filter module were provided to monitor the inlet and outlet pressures of the filter modules. The temperature indicator, TI 1 (FFC1100403700, Anderson Instrument Company, NY) downstream of the cooler monitored the efficacy of the cooling unit. A rotameter, FI 1 (Model U-32477-22, Cole Parmer, IL) downstream of the second filter module measured the flow rate of the feed stream. The permeate recirculation loop consisted of a sanitary centrifugal circulation pump (Model C114-3.81×10^{-2} m × 3.81×10^{-2} m (1.5 in. × 1.5 in.), Waukesha, IL), a back-pulse unit (Model S700-00058, 00914, USF Filtrations and Separations, Fl) and a polished stainless steel (SS 316) permeate tank of 21 capacity (Shickel corp., VA). Two 2.54×10^{-2} m (1-in.) Triclamp diaphragm valves V6 and V13

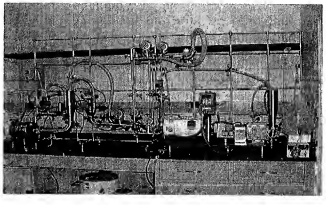

Fig. 3. Ceramic microfiltration pilot plant (vertical pipes in the center contain the ceramic membrane tubes and the feed and permeate reservoirs are on the right and left, respectively). The box on the far-left controls the back-pulsing, while the chiller is on the floor below the table).

(Model 36-2-P10-6L-E-M, Flowtech, GA) located upstream of the permeate ports of each filter module controlled the pressure profile of the permeate circulation system which was monitored by glycerine filled pressure indicators, PI 3–6 (Model EA071010041441, Anderson Instrument Company, NY). The permeation recirculation rate was controlled by throttling valve V7 located in the permeate recirculation pump bye-pass line and monitored by the rotameter, FI 2 (Model U-32477-26, Cole Parmer, IL). Valve V6 was a 2.54×10^{-2} m (1 in.) normally open pneumatically actuated diaphragm valve (Model 36-2-P10-6L-E-AS, Flowtech, GA). This valve closed automatically when the back-pulse valve, BP1 was actuated. BP1 was normally open so that permeate recirculation could occur in the normal direction parallel to the flow of retentate in the filter modules. When the back-pulse occurred, BP1 was closed along with V6. This action trapped the permeate between valves V6 and BP1 while simultaneously forcing a 100 ml quantity of permeate into this segment of piping. This forced the permeate to flow back through the membranes in the reverse direction during a back-pulse. The duration and the frequency of the back-pulse were controlled by the back-pulse control unit. The back-pulse valve was provided with a by-pass arrangement to reduce the back-pulse quantity from 100 ml. This was achieved by a 0.3175×10^{-2} m (1/8 in.) metering valve, V15 (EW-03214-94, Cole Parmer, IL) which by-passed a certain quantity of the trapped permeate back to the permeate reservoir. This quantity could be adjusted by opening V15 suitably. The cooling loop consisted of the previously described shell and tube cooler and a 1 kW glycol chilling unit (Model RS 33A000, KTI, NY) which had a built in pump for recirculation of the glycol water mixture on the shell side of the cooler. Apart from these main components, there are several 0.635×10^{-2} m (1/4 in.) drains and overflow valves attached to the various equipment. The equipment list and the specifications of the main equipment are given in Appendix A.

3.3. IgG assay and yield

This assay was based on a protocol supplied by GTC Biotherapeutics (Framingham, MA). A Protein A affinity chromatography (PA ImmunoDetection^{TM} sensor cartridge (2.1 mm × 30 mm), PerSeptive Biosystems, Framingham, MA) was used to obtain IgG concentrations in the various goat milk streams. Milk samples (1.5 ml) were pipetted into 2 ml Eppendorf centrifuge tubes and centrifuged at 21,000 × g for 30 min. The milk separated into a top fat layer, a clear whey solution and a casein pellet. 0.75 ml of the clear whey phase was carefully extracted with a pipette after puncturing the fat layer. This was pipetted into centrifuge tubes (catalog #8163, Spin-X tubes, Corning, NY) with 0.45 μm pore size cellulose acetate membranes and centrifuged at 21,000 × g for 15 min. The clarified permeate was then injected into the HPLC column. For the permeate samples, sample preparation was unnecessary. A HPLC (Waters 510 with Millennium 2010 operating system) with a 486 UV detector and U6K sample injector were used (Waters Corp., Milford, MA). The loading buffer was 10 mM phosphate buffer, 150 mM NaCl at pH 7.20 ± 0.05 and the elution buffer was 12 mM HCl with 150 mM NaCl. The pump flow rate was set at 2 ml/min and the detector wave length at 280 nm. The injection volume was 10 μl for milk and 20–40 μl for permeate samples. A calibration graph was constructed by injecting different dilutions of IgG fusion protein (GTC Biotherapeutics, Framingham, MA). Loading buffer was passed through the column for 10 min followed by sample injection and loading buffer again for 5 min. After this, elution buffer was run for 10 min. A clean peak corresponding to IgG fusion protein was detected at around 6.5 min into the elution phase. Area obtained by peak integration was compared with the calibration graph to obtain the IgG concentration of the sample after dividing by the sample volume. Care was taken to ensure that all readings were within the range of the calibration graph. This was done by adjusting the sample injection quantities.

The yields for these experiments were computed based on applying a factor of 1.1 to the highest reading of 0.6 g/l obtained from a milk sample with the IgG product. This gave a starting IgG concentration of 0.65 g/l for milk. The yield for a diafiltration experiment was calculated as follows:

$$Y = (N_d\langle Cp\rangle)/(0.65X), \tag{1}$$

where N_d is the number of diavolumes, $\langle Cp\rangle$ is the average permeate IgG concentration and X is the concentration factor of milk prior to diafiltration.

3.4. Protein assay

The Bradford assay (#500-0006, Bio-rad, Hercules, CA) was used to determine protein concentration. This is described in detail in Baruah et al. [7].

3.5. Fat assay

Fat content was measured by the Gerber method which is approved for use by dairies in USA. See Baruah et al. [7] for further details.

3.6. Cleaning regimen

After each experiment, the retentate system was rinsed with deionized water at 50 °C at an axial flow velocity of 4 m/s for 1 min with the permeate ports fully opened. This was followed by recycling cleaning agents Ultrasil 10 – detergent at 0.5 wt.% and Ultrasil 02 – surfactant at 0.1 wt.% at an axial velocity of 4 m/s at 50 °C for 30 min. The loss in volume due to permeation was made up by addition of deionized water at 55 °C. See Baruah et al. for the further details [7]. Thus, the cleaning regimen followed for the pilot plant unit is similar to that for the lab-scale MF unit and gave excellent results. This has tremendous potential for time savings as the acid cleaning step typically used in the dairy industry was not used here [1].

4. Results and discussion

The pilot plant MF experiments were started after the successful optimization of (>90% yield at 5 diavolumes) the lab-scale

Table 1
Pre-optimum results with MF lab-scale unit for IgG (p*I* 7–7.5)

pH	Ionic strength (mM NaCl)	Diavolumes	Flux (lmh)	Yield (%)
6.4	Nil	3	18	16
9	150	3	16	8
6.5	100	3	18	21
7.1	100	2	13	21
7.4	100	3	37	22
7.4	100	5	21	24
7	125	3	10	19
6.8	125	3	10	27
6.6	150	2	9	15
7.1	Nil	4.7	56	45

MF plant with a heterologous IgG product B having a p*I* of 9.0 as reported in Baruah and Belfort [10]. For all the experiments reported here, a different IgG (p*I* 7–7.5) was used. To minimize the amount of milk handled, initial optimization was conducted in the laboratory-scale microfiltration plant as described in [10]. The parameters varied for optimization were pH and ionic strength. The results of these exploratory experiments are tabulated in Table 1. Since results shown in Table 1 were encouraging with product yield reaching 45%, it was decided to conduct the next series of optimization trials at pH 7.1 with no additional NaCl. For this part of the optimization, permeation flux was chosen as the parameter to be optimized. The results of this phase are shown in Fig. 4. At an average permeation flux of 32 lmh, the desired yield of 90% was achieved in 5 diavolumes. After optimizing MF in the laboratory-scale plant, the first phase of optimization experiments were conducted on the MF ceramic pilot plant in the coflow mode without back-pulse, with encouraging results. The salient parameters of the experiments were: (a) milk concentration = 0.64–0.85*X* due to some dilution during start up; (b) milk volume = 2.75 l; (c) pH 7.1; (d) axial velocity = 3.5 m/s; (e) Re = 18,500; (f) UTMP = 3–8.5 psi; (g) diavolumes = 3.25–4.4. The idea was to optimize IgG yield with respect to permeation flux at its p*I* and a moderate axial velocity

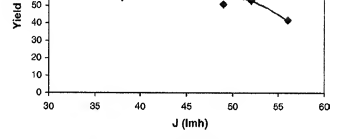

Fig. 4. IgG (p*I* 7–7.5) yield vs. average permeation flux for the short helical hollow fiber module (in the laboratory-scale microfiltration plant with 0.1 μm pore size poly(ether sulfone) hollow fiber membranes) at 1.1 m/s axial velocity (Re ≅ 1075), pH 7.1, 1*X* milk concentration and $N_d = 5$.

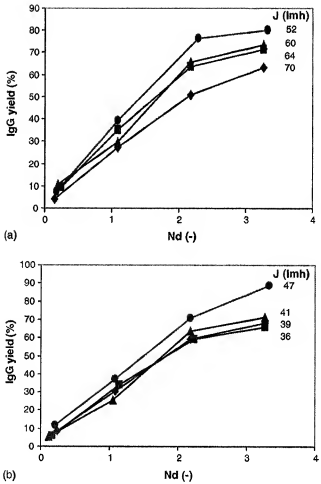

Fig. 5. IgG (p*I* 7–7.5) yield vs. diavolumes at pH 7.1 at a Reynold's number of 18,500 corresponding to an axial velocity of 3.5 m/s and co-flow UTMP operation and milk concentration factors ranging from 0.64 to 0.85*X* for (a) high average permeation flux and (b) low average permeation flux.

of 3.5 m/s (in comparison with the typical value of 7 m/s for the Bactocatch process of Tetrapak) [9].

A series of experiments were conducted with the average permeation flux varying from 36 to 70 lmh (Fig. 5a and b). The highest IgG yield (90% in 3.3 diavolumes) was obtained at an intermediate flux of 47 lmh. The lower yields at permeation fluxes in the region of 36–41 lmh could be due to the lower convection of IgG molecules towards the membrane in these cases. In all cases, the cumulative product yield rose linearly with the number of diavolumes, before tapering off close to 3 diavolumes. The cumulative yields plotted below in Fig. 6 clearly demonstrate that the optimum operating permeation flux is 47 lmh with a corresponding yield of 90% in just 3.3 diavolumes.

The traditional relation between yield Y (in the permeate) and the sieving coefficient S_0 for a diafiltration operation with constant sieving through the membrane is [15]:

$$Y = 1 - \exp(-N_d S_0) \qquad (2)$$

using this, $S_0 = 0.7$. Thus, at the optimum conditions, the sieving coefficient of IgG is a very high figure of 70% at a good average permeation flux of 47 lmh. The corresponding values for the laboratory-scale MF unit are 46% and 32 lmh. Hence,

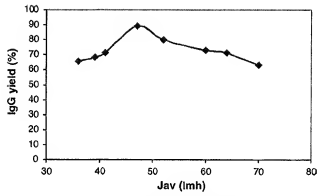

Fig. 6. IgG (p/ 7–7.5) yield after 3.3 diavolumes vs. average permeation flux, at pH 7.1 at a Reynold's number of 18,500 corresponding to an axial velocity of 3.5 m/s and co-flow UTMP operation and milk concentration factors ranging from 0.64 to 0.85X.

the ceramic pilot plant gives a 50% improvement over the laboratory-scale MF unit both in product transmission through the membrane and in the permeation rate.

Several experiments were conducted at higher axial velocities and back-pulsing at a frequency of once in 5 min. With these experiments, permeation flux increased to around 60 lmh, but the yields were reduced to 80%. It appears that back-pulsing is not advantageous for this particular application with milk. However, it holds promise for other applications [11–13].

5. Conclusions

A highly instrumented pilot plant was designed, built and tested with excellent results (90% yield of IgG at 47 lmh) with a complex polydisperse suspension like transgenic whole goat milk. This ceramic plant combines desirable features such as a hydrophilic membrane of high durability and resistance to heat and chemicals, with excellent fluid mechanics resulting from turbulent flow and uniform transmembrane pressure. Specifications and design calculations are described in detail in the Appendices A and B, respectively. To our knowledge, this represents a unique combination of features and makes the pilot plant versatile enough to tackle various challenges of MF encountered in the food processing, biotechnology, pharmaceutical, beverages and other chemical processing industries.

Acknowledgements

GTC Biotherapeutics (Framingham, MA) is thanked for funding the work and for supplying the transgenic goat milk. Special thanks are due to Daniel Couto, Estzer Birck-Wilson and Wesley Church for the product data, the Protein A affinity chromatography column and protocol and IgG standards. Millipore Corporation, Bedford, MA is thanked for supplying the hollow fiber modules for the laboratory-scale MF plant and Ecolab, St. Paul, MN for the detergent and surfactant. Robert van Reis (Genentech, CA), Richard Mcdonnough (Memtech, CA), Gunnar Jonsson (Danish Technical University, Lyngby, Denmark) and Niels Ottosen (DSS Silkeborg AS, Denmark) are thanked for their advice and suggestions.

Appendix A. Ceramic pilot plant specifications

- Process specification of ceramic filtration system—CF00 Rev. 0 (see Table A.1)
  - Filter type: tubular ceramic (removable/replaceable preferred)
  - Pore size: 0.2 μm
  - Filtration area: 0.03 m^2
  - System hold up volume: 2.5 l max inclusive of filter, pump, reservoir and piping
  - Minimum instrumentation:
    - (a) Temperature gauge: 1
    - (b) Press gauges: 5 (inlet/outlet of tube side, pump discharge and permeate)
    - (c) Flow meters: 1
    - (d) By-pass arrangement for pump
  - Feed pump: capable of generating a tube side velocity up to 7 m/s
  - Additional features required: (a) co-flow system of permeate on shell side to maintain constant TMP, (b) periodic

Table A.1
Equipment list for ceramic MF pilot plant

Tag no.	Item	Specification
Equipment list		
F1	Ceramic filter(with co-flow)	CF01
P1	Feed pump	CF02
P2	Permeate circulation pump	CF03
P3	Cold water circulation pump	CF04
H1	Milk cooler	CF05
H2	Chiller	CF06
T1	Retentate tank (SS)	CF07
T2	Permeate balance tank (SS)	CF08
T3	Permeate collection flask	CF09
PI1	Pressure indicator	CF10
PI2	Pressure indicator	CF10
PI3	Pressure indicator	CF10
PI4	Pressure indicator	CF10
PI5	Pressure indicator	CF10
TI1	Temperature indicator	CF11
FI1	Rotameter with valve	CF12
FI2	Rotameter with valve	CF12
V1	1/2 in.	CF13
V2	1/2 in. diaphragm valve	CF14
V3	1/2 in. diaphragm valve	CF14
V4	1/2 in. diaphragm valve	CF14
V5	1/2 in. needle valve	CF13
V6	1/2 in. actuated diaphragm valve	CF17
V7	1/2 in. diaphragm valve	CF14
V8	1/2 in. needle valve	CF13
V9	1/2 in. needle valve	CF13
V10	1/2 in. needle valve	CF13
V11	1/2 in. needle valve	CF13
V12	1/2 in. needle valve	CF13
V13	1/2 in. diaphragm valve	CF14
V14	1/2 in. diaphragm valve	CF14
V15	1/8 in. metering valve	CF18
Spare	1/2 in. needle valve	CF13
Spare	1/2 in. diaphragm valve	CF14
BP1	Back pulse unit	CF15
	Pipes and fittings	CF16
	Installation hardware	

back-pulsing of permeate to feed side, (c) temperature control
  - Mode of operation: diafiltration of biofluid
- Feed pump—CF02 Rev. 3 (Fig. 2)
  - Tag number: P1
  - Type: rotary positive displacement with variable speed
  - Construction: sanitary construction with SS 316L wetted parts
  - Flow rate: 1.1 m^3/h
  - Discharge pressure: 7 bar
  - Service: Milk at 3 cp
  - Make: Waukesha
  - Model: U1 018
- Permeate circulation pump: CF03 Rev. 3 (Fig. 2)
  - Tag number: P2
  - Type: centrifugal
  - Construction: sanitary construction with SS 316L wetted parts
  - Flow rate: 15 gpm
  - Discharge pressure: 100 feet (3 bar)
  - Service: permeate (consider properties as for water)
  - Make: waukesha
  - Model: C216
- Cooler—CF05 Rev. 3 (Fig. 2)
  - Tag number: H1
  - Type: shell and tube
  - Construction: SS 316L, sanitary
  - Rating: 1 kW
  - Design pressure: 10 bar
  - Milk flow rate: 1.1 m^3/h
  - Water flow rate: 0.7 m^3/h
  - Milk inlet temperature: 23 °C
  - Milk outlet temperature: 20 °C
  - Cooling water inlet temperature: 15 °C
  - Cooling water outlet temperature: variable
  - Milk side pressure drop: 0.15 bar
  - Water side pressure drop: variable
  - Make: Exergy
  - Model: 00455-1
- Chiller—CF06 Rev. 2 (Fig. 2)
  - Tag number: H2
  - Construction: SS
  - Rating: 1000 W
  - Service: water
  - Type: recirculating
  - Make: KTS, Model: RS33A000

Appendix B. Calculations for the ceramic pilot plant

1. Ceramic filter
   - Main process requirement: ability to process 7 diavolumes in 4 h.
   - System volume: 0.5 l
   - Processing volume: 3.5 l
   - Based on previous experiments, J: 35 lmh
   - Area of filtration required: $3.5/(35 \times 4) = 0.025\ m^2$
   - Considering US Filter system, we select 7 mm ID channel element. For this element filtration, area = $0.005\ m^2$ for a length of 250 mm. Hence,
   - Desired length = $(0.025/0.005) \times 250\ mm = 1250\ mm$
   - We select a length of 1500 mm to give a 20% margin in filtration area. Hence, the filter parameters are:
   - Pore size: 0.2 μm
   - Channel ID: 7 mm
   - Channel length: 1500 mm
   - Number of channels: 1
2. Pressure drop in ceramic module [16]
   Formula used: $F = (4fL/D)(v^2/2g)$ (5.52)
   where, f (friction factor) is calculated based on Moody' chart Fig 5.26 [16]. Data: $L = 1.5$ m; $D = 0.007$ m; $V = 7$ m/s; $g = 9.81\ m/s^2$; $\varepsilon = 0.001$ feet (for smooth concrete from Table 5-7); $\mu = 1$ cp $= 0.001$ kg/(ms); $\rho = 1000\ kg/m^3$.
   Calculations:
   (a) Tube side
   - Filters

   $$Re = \rho Dv/\mu = 1000 \times 0.007 \times 7/0.001$$
   $$= 49000 \text{ for } v = 7 \text{ m/s}$$

   $\varepsilon/D = 0.001 \times 25.4 \times 12/7 = 0.044$
   From Fig 5.26, $f = 0.018$, pressure drop, $F = (4 \times 0.018 \times 1.5/0.007) \times (7 \times 7/2 \times 9.81) = 38.5$ m liquid column = 3.85 bar for $v = 7$ m/s.
   - Cooler
     For the model Exergy 00455-1, the pressure drop on the tube side at 1.1 m^3/h (4.8 gpm) is 2 psi or about 0.14 bar.
   - Piping (1.27×10^{-2} m (0.5 in.))
     $v = 2.2$ m/s for 1.27×10^{-2} m (0.5 in.) piping (see Section 5 below). Pipe length = 2 m, 8–90° elbows($K = 0.75$) and 1-diaphragm valve ($K = 2.3$). K values for fittings are taken from Table 5-19. $Re = \rho Dv/\mu = 1000 \times 0.0125 \times 2.2/0.001 = 27500$; from Fig 5.26, $f = 0.006$ for smooth pipes. Pressure drop, $F = (4fL/D + \sum K)(v^2/2g) = (4 \times 0.006 \times 2/0.0125 + 0.75 \times 8 + 2.3) \times (2.2 \times 2.2/2 \times 9.81) = 2.99$ m liquid column = 0.3 bar. Hence, the total pressure drop = 3.85 + 0.3 + 0.3 = 4.45 bar.

   (b) Shell side
   To maintain the same pressure drop in the shell side, we select the same velocity as the tube side that is, 2 m/s. According to USF (telecon with Rishi on 2/21/02) tube OD = 10 mm and shell ID = 1.587×10^{-2} m (0.625 in.) = 15.875 mm max. Therefore, the annular area = $\pi/4(15.875^2 - 10^2) = 119\ mm^2$. Required flow rate = $(119/1000000) \times 7 \times 3600\ m^3/h = 3\ m^3/h = 13.21$ gpm
3. Pumps
   (a) Feed pump (P1)
   To be designed for a velocity of 7 m/s in the tube; $Q = (\pi/4)(7/1000)^2 \times 7 \times 3600\ m^3/h = 0.97\ m^3/h$; we choose $Q = 1.1\ m^3/h$. Based on our pressure drop

calculations, the total pressure drop on the feed circuit = 4.45 bar. We select a pump head = 7 bar to give adequate margin.

(b) Permeate pump (P2): Based on USF data, $Q = 13.24$ gpm, we choose $Q = 15$ gpm with pump head of 3 bar.

4. Cooler (H1) and chiller (H2)

Feed pump circulation heat = $(dm/dt)gh/\eta$. Selecting a low pumping efficiency of 0.25, we get $Q = (1100 \times 9.81 \times 70 \times 1/3600)/0.25 = 839$ W. We select 1000 W for the chiller that generates cold water and 1000 W for the cooler that cools milk.

5. Piping

- Feed piping:

  To minimize pressure drop, we consider 1.27×10^{-2} m (1/2 in.) piping. This will give a velocity of $(7/12.5)^2 \times 7 = 2.2$ m/s which is reasonable.

- Permeate piping:

  We consider a velocity of 2 m/s for a flow rate of 3 m^3/h. Cross sectional area required = $3/(2 \times 3600) = 0.00042\ m^2$. Hence, ID = $((0.00042 \times 4)/\pi)^{0.5} = 23$ mm. We select 2.54×10^{-2} m (1 in.) piping.

- Drain piping:

  We select 0.635×10^{-2} m (1/4 in.) tubing as vessel hold-up volumes are all in the range of 1 l only.

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